

# PATENT SPECIFICATION



917,947.

Date of Application and filing Complete Specification:  
May 10, 1960. No. 16506/60

Complete Specification Published: February 13, 1963

Index at Acceptance:—Classes 1(3), A1D12, A1G(24D12:39D12:46D12:51D12), A1GX12,  
A1N(4B:10A:13A:24A:34:39B), A1NX2; and 95, A5.

International Classification:—C01f, g, C09d.

## COMPLETE SPECIFICATION

### NO DRAWINGS

#### Process for the Preparation of Organophilic Oxides and Chromate Pigments

We, LAJOS CSONKA, of 14. Pazsit utca,  
Budapest II, Hungary, FERENC HORKAY, of  
4, Kapitany utca, Budapest XII, Hungary,  
FERENC SZANTO, of 6, Corvin utca, Budape-  
st IV, Hungary, JANOS SZERECZ, of 8,  
Guyon koz, Budapest II, Hungary, and  
JENO GONCZY, of 34. Csalan utca, Budape-  
st II, Hungary, all Hungarian citizens, do  
hereby declare the invention, for which we  
pray that a patent may be granted to us, and  
the method by which it is to be performed,  
to be particularly described in and by the  
following statement:—

This invention relates to the preparation of  
organophilic pigments.

It is usual to treat lithopone pigments with  
water-soluble soaps in order to improve  
their mixing and dispersing properties.  
Lithopone, which is known to be a mixture  
of zinc sulfide and barium sulfate, can be  
thus made organophilic to a satisfactory ex-  
tent. The same process is, however, not suit-  
able for oxide pigments, since on the addi-  
tion of the soap solution they will form a  
highly dispersed suspension. Oxide pigments  
do not precipitate and thus cannot be pro-  
cessed for industrial use. Chromate pigments  
change their color upon the addition of the  
soap, whereby their quality is deteriorated.

We have found that the above drawbacks  
may be removed by treating the pigment sub-  
stance with anionic surface active agent, and  
subsequently acidifying the solution, prefer-  
ably to pH-5. Thereupon the oxide pigments  
become readily filterable, and easily remov-  
able from the liquid. After washing,—if  
necessary—they are dried and, if desired, dis-  
integrated. After acidifying, chromate pig-  
ments recover their original color so that ex-  
cellent quality organophilic and readily filter-  
able pigments are obtained. The organo-  
philic pigments thus obtained are highly  
dispersible in organic solvents and can be

readily processed to stable suspensions.

According to the invention there is pro- 45  
vided a process for rendering pigments  
selected from oxide, hydrated oxide and  
chromate pigments organophilic which com-  
prises reacting an aqueous suspension of the  
pigment with a solution of an anionic surface 50  
active agent, acidifying the solution, separat-  
ing the pigment from the liquid, then drying  
and, if desired, disintegrating the product  
thus obtained.

Mineral acids, e.g. hydrochloric acid, 55  
sulphuric acid and phosphoric acid, are pre-  
ferably used to acidify the aqueous medium;  
however, strong organic acids, e.g. acetic  
acid may also be used.

Suitable anionic surface active agents for 60  
use in the process according to the invention  
include alkali metal and ammonium salts of  
saturated or unsaturated fatty acids having  
at least 8 carbon atoms, preferably having  
from 16 to 18 carbon atoms. Other surface 65  
active agents which may be used are the  
alkali metal or ammonium salts of fatty  
acid containing an OH group, for example  
ricinoleic acid. There may also be used  
alkali metal or ammonium salts of partly 70  
polymerised unsaturated fatty acids as well  
as alkali metal and ammonium salts of in-  
dustrial stearine, comprising a mixture of  
stearic, palmitic and oleic acids.

Another class of surface active agents 75  
which may be employed as anionic surface  
active agents are the alkali metal salts of  
alkyl sulphonic, aryl sulphonic and mixed  
alkyl-aryl sulphonic acids.

The pigments used in the process accord-  
ing to the invention are oxide or hydrated  
oxide pigments precipitated or prepared by  
pyrogenic processes, for example titanium  
dioxide, red and yellow iron oxides, green  
chromium oxide, red bauxite (obtained by 80  
heating banxite) and aluminium oxide and

chromate pigments, for example zinc chromate, zinc tetroxy chromate, lead chromate and basic lead chromate. These pigments have positive surface charges in aqueous 5 solution and are capable of ion exchange when reacted with anionic surface active agents. The surface active agent ions adsorbed on the surface of the pigment particles become irreversibly bound to the surface upon subsequent acidification and drying following upon filtering. Thus, an organophilic film fixed on the surface of the said 10 pigment particles is produced, whereby the dry pigment particles exhibit an organophilic 15 character.

In the case of chromate pigments the optimum amount of the substance rendering them organophilic can be established by experimental trials. It has been found that the 20 amount depends on the kind and particle size of the pigment. It is advisable to carry out the trials in the following manner: A series of equal amounts of pigment are suspended in equal amounts of water, then equal volumes 25 of aqueous solutions are added containing increasing amounts of the anionic surface active agents. The suspensions are thoroughly agitated, and after a certain time the volume of the sediment is ascertained. The 30 largest amount of sediment, that is, the amount of the surface active agent associated therewith will indicate the optimum amount. The pigments treated with the amount so established will exhibit the best organophilic properties and will unite most readily and with the highest dispersion with the 35 organic substance of the paint vehicles where they form non-settling stable suspensions.

The step of rendering the pigment substances organophilic can be most conveniently effected as follows: 10 to 50% pigment is suspended in water, and a 1 to 4% aqueous solution, heated preferably to 70 to 80°C, of the surface active agent is added thereto at 40 an elevated temperature, preferably at 70 to 80°C. 0.5 to 5% of the organophilizing agent, depending on the experimental trial, based on the pigment weight, is used. The pigment suspension so prepared is then acidified to 45 pH 5, then the pigments are readily separated from the water, washed, dried preferably at 60 to 100°C and disintegrated.

*Example 1.*  
500 g titanium dioxide are suspended in 55 2 litres of distilled water, and 1 litre 1% ammonium stearate is added while stirring. Stirring is carried on, and diluted hydrochloric acid is used for adjusting the pH to 5. The pigment suspension thereupon forms a 60 readily filterable precipitate, which is filtered off, washed if necessary with water, dried at 80 to 100°C, then granulated in an edge runner. The pigment so obtained is once milled in a three-roller mill and is used for preparing 65 an enamel paint which is still entirely

free from sediments after one year. Subjected to a weather exposure test, it showed a life thrice as long as an enamel paint containing non-organophilic pigments. It has better spreading properties and a higher gloss and 70 the films obtained therefrom are highly water-proof and the pigment particles will not migrate to the surface.

Organophilic pigments can be produced in the same manner from iron oxide yellow 75 and iron oxide red, chrome oxide green, aluminium oxide and bauxite red.

*Example 2.*

Experimental trials showed that the best addition to a lead chromate pigment in an 80 aqueous suspension is 1% by weight of ammonium stearate, as the latter produced the largest sediment volume. 500 g lead chromate are suspended in 2 litres of water, then 500 g of 1% ammonium stearate are 85 added at 65 to 70°C under brisk agitation. Thereupon the light yellow pigment turns orange. The pH of the pigment suspension is adjusted with hydrochloric acid to 5, whereupon the chrome yellow regains its original 90 colour. The precipitate is filtered, washed with water, dried at 60 to 100°C and homogenised in an edge runner. The ammonium salts of fatty acids have the advantage over sodium soaps that upon acidification the 95 original colour shade can more preferably be reestablished. The NH<sub>4</sub>Cl which is formed can be driven off in course of drying.

Organophilic pigments can be prepared likewise from zinc chromate, zinc tetroxychromate, barium chromate, or strontium chromate. 100

*Example 3.*

The organophilic pigment obtained in accordance with Example 1 or 2 can be readily 105 employed in non-polar or slightly polar organic media and lacquer base materials, for example oil varnish or oleoresinous varnish, solutions of epoxy resins esterified with fatty acids, oil modified alkyd resin solutions. 110

Organophilic pigments of good wetting properties which can be suspended in polar organic media, such as alcohols, ketones, esters, nitrocellulose lacquers, spirit varnishes, alkyd-urea-formaldehyde vehicles, and 115 alkyd melamine-formaldehyde vehicles can be obtained with the process described in the preceding examples, but using, instead of ammonium stearate, the equivalent amount of surface active agent derived from a fatty 120 acid containing an OH-group, for example ammonium ricinoleate.

**WHAT WE CLAIM IS:—**

1. A process for rendering pigments selected from oxides, hydrated oxide and 125 chromate pigments organophilic which comprises reacting an aqueous suspension of the pigment with a solution of an anionic surface active agent, acidifying the solution, separating the pigment from the liquid, then 130

drying and, if desired disintegrating the product thus obtained.

2. A process according to claim 1, wherein the aqueous solution is acidified to pH 5.

5. 3. A process according to claim 1 or claim 2, wherein a mineral acid is used for the acidification.

4. A process according to any of claims 1 to 3, wherein the anionic surface active agent is an alkali metal or ammonium salt of a fatty acid having at least 8 carbon atoms.

10. 5. A process according to claim 5, wherein the fatty acid has from 16 to 18 carbon atoms.

15. 6. A process according to claims 1 to 3, wherein the anionic surface active agent is an alkali metal or ammonium salt of a fatty acid containing an OH group.

7. A process according to claim 6 where-  
20 in said fatty acid is ricinoleic acid.

8. A process according to any of claims 1 to 3, wherein the anionic surface active agent is an alkali metal or ammonium salt of industrial stearine.

9. A process according to any of claims 25 1 to 3, wherein the anionic surface active agent is an alkali metal or ammonium salt of a partly polymerised unsaturated fatty acid.

10. A process according to any of claims 30 1 to 3 wherein the anionic surface active agent is an alkali metal salt of an alkyl sulphonate acid, aryl sulphonic acid or mixed alkyl-aryl sulphonic acid.

11. A process for the preparation of 35 organophilic pigments substantially as herein described with reference to any of the Examples.

12. Organophilic pigments whenever prepared by a process as claimed in any of the 40 preceding claims.

For the Applicants.  
FRANK B. DEHN & CO.,  
Chartered Patent Agents,  
Imperial House, 15-19 Kingsway,  
London, W.C.2.

Berwick-upon-Tweed: Printed for Her Majesty's Stationery Office by The Tweeddale Press Ltd.—1963  
Published at The Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may  
be obtained.